

FORM PTO-1390
(REV 10-97)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

DATE: June 21, 2000

EXPRESS MAIL LABEL NO.
EL496185049USATTORNEY DOCKET NO.
39629/DBPU.S. APPLICATION NO.
Be Assigned
097582175INTERNATIONAL APPLICATION NO
PCT/IB98/01039INTERNATIONAL FILING DATE
7. July.1998PRIORITY DATE CLAIMED
23.December.1997TITLE OF INVENTION
ION EXCHANGE SYSTEM USING U-TUBE PRINCIPLEAPPLICANT(S) FOR DO/EO/US
Arianto Darmawan

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/LUS)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items below concern other document(s) or other information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Small entity claim with a copy of this transmittal letter attached.
17. ☒ International search report.
18. ☒ International preliminary examination report.
19. ☒ Extra set of Drawings
20. ☒ Letter Regarding Typographical Error
21. ☐

ORIGINAL RECEIVED

U.S. APPLICATION NO. (If none, see 37 CFR 1.5) To Be Assigned 09/582175		INTERNATIONAL APPLICATION NO. PCT/IB98/01039		ATTORNEY DOCKET NO. 39629/DBP							
<input checked="" type="checkbox"/> The following fees are submitted: (see Note (1) below) Basic National Fee (37 CFR 1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO \$ 840.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) ... \$ 670.00 No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$ 690.00 Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO. \$ 970.00 International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$ 96.00 <div style="text-align: right; margin-top: 10px;">ENTER APPROPRIATE BASIC FEE AMOUNT =</div>				<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 50%;">CALCULATIONS</th> <th style="width: 50%;">PTO USE ONLY</th> </tr> <tr><td style="height: 100px;"></td><td></td></tr> </table>		CALCULATIONS	PTO USE ONLY				
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Claims	Number Filed	Number Extra	Rate								
Total Claims	5 -20=	0	X \$18	\$ 0.00							
Independent Claims	2 -3=	0	X \$78	\$ 0.00							
Multiple dependent claim(s) (if applicable)			+ \$260	\$ 0.00							
TOTAL OF ABOVE CALCULATIONS =				\$ 840.00							
Reduction by 1/2 for filing by small entity, if applicable. Verified Small entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				<table border="1" style="width: 100%; border-collapse: collapse;"> <tr><td style="height: 30px;"></td><td></td></tr> </table>							
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Processing fee of \$130 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				<table border="1" style="width: 100%; border-collapse: collapse;"> <tr><td style="height: 30px;"></td><td></td></tr> </table>							
TOTAL NATIONAL FEE =				\$ 420.00							
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				<table border="1" style="width: 100%; border-collapse: collapse;"> <tr><td style="height: 30px;"></td><td></td></tr> </table>							
TOTAL FEES ENCLOSED =				\$ 420.00							
Note (1): The basic national fee must be paid when filing this application. The 20-month time limit (37 CFR § 1.494) and 30-month time limit (37 CFR § 1.495) are not extendable.				<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; text-align: right;">Amount to be:</td> <td style="width: 50%;"></td> </tr> <tr> <td style="text-align: right;">refunded</td> <td style="text-align: center;">\$</td> </tr> <tr> <td style="text-align: right;">charged</td> <td style="text-align: center;">\$</td> </tr> </table>		Amount to be:		refunded	\$	charged	\$
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<p>a. <input checked="" type="checkbox"/> A check in the amount of \$ <u>420.00</u> to cover the above fees is enclosed.</p> <p>b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>03-1728</u>. A duplicate copy of this sheet is enclosed.</p> <p>NOTE (2): Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p> <p>SEND ALL CORRESPONDENCE TO:</p> <div style="display: flex; justify-content: space-between; margin-top: 20px;"> <div style="width: 45%;"> <p>D. Bruce Prout CHRISTIE, PARKER & HALE P.O. Box 7068 Pasadena, CA 91109-7068</p> </div> <div style="width: 45%; text-align: right;"> <p>By <u><i>D. Bruce Prout</i></u> D. Bruce Prout Reg. No. 20,958</p> </div> </div>											

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CHRISTIE, PARKER & HALE, LLP

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Entitled : ION EXCHANGE SYSTEMS USING U-TUBE PRINCIPLE

VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS

(37 CFR § 1.9(f) and § 1.27(b))

INDEPENDENT INVENTOR

As a below-named inventor, I declare that I qualify as an independent inventor as defined in 37 CFR § 1.9(c) for purposes of paying reduced fees under Sections 41(a) and (b) of Title 35, United States Code, to the Patent and Trademark Office with regard to the invention entitled

ION EXCHANGE SYSTEMS USING U-TUBE PRINCIPLE

described in

X the specification filed herewith
application Serial No. _____, filed _____
Patent No. _____, issued _____

I have not assigned, granted, conveyed or licensed, and am under no obligation under contract or law to assign, grant, convey or license, any rights in the invention to any person who could not be classified as an independent inventor under 37 CFR § 1.9(c) if that person had made the invention, or to any concern which would not qualify as a small business concern under 37 CFR § 1.9(d) or a nonprofit organization under 37 CFR § 1.9(e).

Each person, concern or organization to which I have assigned, granted, conveyed, or licensed, or am under an obligation under contract or law to assign, grant, convey, or license any rights in the invention is listed below.

X No such person, concern or organization.
Persons, concerns or organizations listed below. (NOTE: Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. 37 CFR § 1.27)

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X INDIVIDUAL _____ SMALL BUSINESS CONCERN _____ NONPROFIT ORGANIZATION

Full name _____

Address _____

_____ INDIVIDUAL _____ SMALL BUSINESS CONCERN _____ NONPROFIT ORGANIZATION

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR § 1.28(b))

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

Arianto DARMAWAN

15 May 2000

NAME OF INVENTOR

SIGNATURE

DATE

NAME OF INVENTOR

SIGNATURE

DATE

NAME OF INVENTOR

SIGNATURE

DATE

Description

AN ION EXCHANGE SYSTEM WITH COMPACTED MEDIA
DURING OPERATION AND REGENERATION
PROCESSES USING U-TUBE PRINCIPLE

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Field of Invention

This invention is concerned with processes of liquid separation using ion exchange system and in particular forms is related to method for demineralizing or softening of water, purification of aquaous solution, recovery of metals and valuable compounds.

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Background of Invention

Ion exchange system is a process of liquid separation. Generally this process uses ion exchange resin, adsorbent resin or catalytic resin.

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Ion exchange resins are polymeric beads, granulas or powders having a functional acidic group called cation resin and basic group called anion. They work by exchanging ions in a solution with ions fixed inside the porous polymer matrix of the resin. Adsorbent resins are more porous and work by attracting materials onto the large surfaces of the resin. Catalytic resins are polymer beads or powders with acidic or basic groups and provide an effective, environmentally friendly alternative to liquid catalyst.

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In most cases involving ion exchange system the resin is confined in a column. The system may contain one or more columns filled with one or more types of resin of the same or different function. The particular advantage of such ion exchange liquid separation systems as described above lies in their effectiveness, high efficiency, simplicity and low operating cost compared to other separation processes such as evaporation, reverse osmosis and the like. Successful liquid separation processes using ion exchange system depend in major part, upon the characteristic of the system utilized. Among the desired characteristics are:

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- High purity of the processed product.

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The system must be able to ensure production of high quality of demineralized or adsorbed product. It should keep the resin bed in a compacted form and reduce the ion leakage.

- Low chemical consumption
Regeneration of the exhausted resin should be performed efficiently for optimum chemical consumption and should maximize the resin bed operating capacity such as to produce the most highly regenerated resin at the service flow outlet.
 - Less waste during regeneration
Regeneration is to be performed efficiently, less time, less water consumption for backwashing and rinsing, and less chemical waste.
 - Reduced pressure drop
High flow rate increases pressure drop. High pressure drop increases possibility of breakage of the resin beads and causes higher pumping energy.
 - Wide range of flow rates
Since most processes are not constant the system should be operable for processes at wide range of flow rates and is still capable to produce highly purified product.
 - Low operating and investment cost
The system should be easily operable with low operating, investment and maintenance costs.
- The following basic ion exchange systems are generally available.
- The first system is a cocurrent system where the direction of flow during service is from top to bottom while the regeneration process is also from top to bottom (DEGREMONT. WATER TREATMENT HANDBOOK, 5th EDITION, JOHN WILEY & SONS 1979 PAGE 319-320). In this system the resin bed is compacted during service and regeneration. During service the liquid is passed through a column which contains resin beads supported by underdrain which normally contains nozzles at the bottom of the tank. There is a free board above the surface of the resin almost as high as the resin bed to allow the resin bed to be effectively backwashed before the regeneration process to clean and remove the small broken particles.

This cocurrent system is widely used since it is simple but requires plenty of regenerant. Because the regenerant flow is from top to bottom the highly regenerated resin bed is at the service flow inlet while the lower regenerated resin bed is at the service flow outlet. Therefore the purity of the processed products is not very high. This system requires a lot of regenerant and should be backwashed before each regeneration process.

The second system is a counter current system where the direction of flow during service is from top to bottom while the flow of the regeneration process is from bottom to top (THE UPCORE SYSTEM, DOW CHEMICAL COMPANY, FORM No.17701527895 CH 17280 E 0895 R). The upper part of the column is equipped with upper bed containing nozzles identical to that of the underdrain bed. There is a free board above the resin bed to accommodate 5-10 % expansion of the resin bed after being exhausted.

The disadvantage of this system is that during the regeneration process, where the regenerant flow is from bottom to top, the resin will not be fully compacted due to the tendency of fluidization which causes the regeneration process less effective. Because of the low upflow speed of the limited quantity of regenerant as well as the tendency of the resin bed to fall down due to the higher specific gravity of the resin as compared to that of the liquid, more regenerant are required during regeneration although it is still less than that of the cocurrent system.

Tank diameter is correlated to the upflow speed of the regenerant. Increasing the upflow speed would result in smaller tank diameter and smaller resin volume and therefore shorter regeneration cycle.

There is no backwashing facility and the resin should be discharged from the column if the pressure drop of the system exceeds the limit due to the accumulation of the broken resin particles on the nozzles opening. This system requires specially sized resin with more uniform particles to reduce pressure drop.

The third system is a counter current system where the direction of flow during service is from bottom to top and the regeneration process is conducted from top to bottom (AMBERPACK BACKWASHABLE PACKED BED SYSTEM [EXTERNAL BACKWASHING] ROHM AND HASS; PRINT INF 9003 A DEC 93 IMPACTES RCS B 350424636). The upper part of the

column is equipped with upper bed which contains nozzles identical to that of the underdrain bed. There is a free board above the resin bed to accommodate 5% to 10 % expansion of the resin bed after being exhausted.

5 The disadvantage of this system is that during service, where the flow is from bottom to top, the flow speed must be relatively high in order to obtain a compacted resin bed. In many operations the service flow rate is fluctuating or interrupted which causes fluidization or fall of the resin bed. This makes the resin bed uncompacted and contaminates the highly
10 regenerated resin at the flow outlet which will reduce the water quality and exchange capacity.

Small broken resin particles tends to rise to the top and resin fines with the size smaller than the nozzle openings may be transported and contaminate the other ion exchange vessel. To protect the system a resin
15 trap should be installed, but it will increase the pressure drop. There is no internal backwashing facility and the resin should be discharged to be backwashed outside of the column if the pressure drop of the system exceeds the limit due to the accumulation of the broken resin particles having sizes larger than the nozzle openings. This system requires specially
20 sized resin with more uniform particles to reduce pressure drop.

US Patent 1 688 915 (year: 1928) utilizes two connected compartments as apparatus for treating liquids. This invention emphasized that no screen are required for carrying the weight of the softening material because the weight of the softening material is carried directly by the tanks
25 or containers themselves. No compaction of the softening material and counter current effect was intended.

One can understand this phenomenon since at the time this invention was filed, softening material contain mostly of minerals which are much heavier than nowadays resin. This system does not have a separate
30 backwashing port.

The tank diameter is correlated with the upflow speed during service where increasing the speed requires smaller tank diameter and therefore yields smaller resin volume and consequently shorter regeneration cycle.

The fourth system is a counter current system with water or air hold
35 down (COUNTER CURRENT ION EXCHANGE SYSTEMS IN INDUSTRIAL

AND UTILITY WATER TREATMENT. V.R. DAVIES. ROHM AND HAAS COMPANY, MARCH 1989). This system is a down flow service and upflow regeneration. In the water hold down system, there is a free board above the resin bed with distributor at the top. Regenerant exit distributor is buried just below the upper surface of the resin bed.

During regeneration cycle, the regenerant will flow upward from the bottom and exit through this collector while at the same time water will flow downward from the distributor through this exit distributor to maintain packed resin bed. In the air hold down system, air is used instead of water for the regeneration process.

The disadvantage of this system is that it requires a relatively large quantity of water or air during regeneration to compact the resin bed. Besides, the operation and maintenance are more complicated.

Counter current system shows advantages over cocurrent system. During regeneration process the resin bed at the regenerant inlet will be highly regenerated with the highest exchange capacity while the resin bed at the regenerant outlet will remain partially in the exhausted stage and has lower exchange capacity. In the cocurrent system where the service inlet is the same as the regenerant inlet, this partially exhausted stage will affect the leakage influencing the quality of the treated liquid. It is possible to increase the quality of the treated liquid by imposing higher regeneration level to convert the partially exhausted stage resin to become more highly regenerated but it will be less economical.

In the counter current system the outlet liquid will be in contact with highly regenerated portion of the resin during the service which yields much higher quality of processed liquid with less leakage and less regenerant cycle.

Summary of invention

The principal objective of this invention is developing a design of an ion exchange system for producing the highest purity of processed product with the following features: low chemical consumption, easy operation and maintenance, less waste, low operating cost, low investment cost and has a wide range of application.

Another objective of this invention is developing a design of a counter

current ion exchange system which can tolerate changes of flow rates during service cycle or even interruption of service flow. Furthermore the system should have the capability of internal backwashing whenever required to remove the resin fines if the pressure drop exceeds the expected limit. The system can be operated at low service flow rate with low pressure drop so that besides specially sized resin, standard sized resin may be used.

The aforementioned principal objective has been achieved by building a system operating on a counter current base where the resin is always compacted during service cycle, regenerated cycle or even when the flow is interrupted. The system is also provided with backwashing facility where the backwashing flow rate can be adjusted. The backwashing is in effect only if the pressure drop of the system increases such as to exceed a certain limit. In normal condition backwashing is required only after more than one hundred regeneration cycles, so there is practically no backwashed waste water in the regeneration process except once a year or whenever required.

The design of the system of this invention comprises a vertical column divided into two vertical compartments with free space in the lower part so that both compartments are interconnected and form a U tube type connection. They are filled with one or more types of ion exchange resin. The upper part of each compartment is equipped with upper bed containing nozzles. There is a free board above the resin bed to accommodate the expansion of the resin bed occurring after being exhausted or during regeneration. The direction of flow during service cycle is from top to bottom in one compartment and upward in the other compartment. The direction of flow during regeneration process, which is conducted from the other compartment, is also from top to bottom and then flows upward through the other side.

One configuration is a vertical column usually round in shape with vertical partition inside which divides this column into two vertical compartments with free space at the lower part so that it forms a U tube type connection.

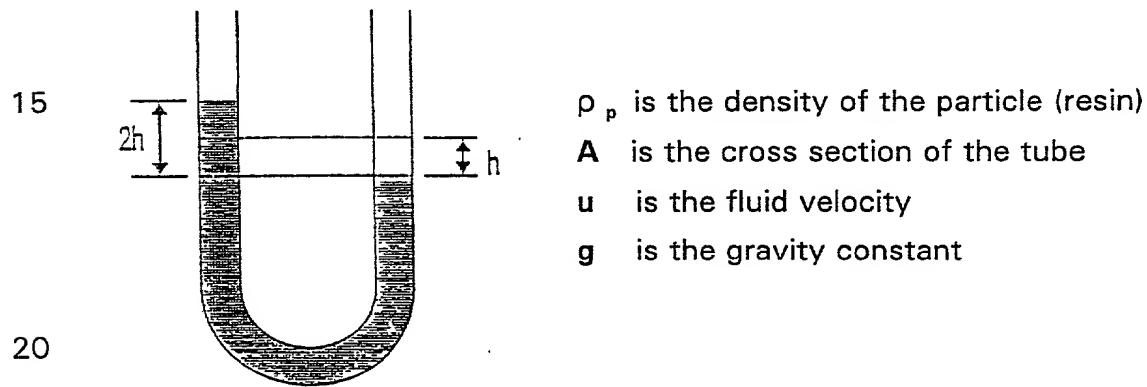
The second configuration is a vertical column usually round in shape with another vertical column located concentrically inside. There is a free

space interconnecting both columns at the bottom part so that they form a U tube type connection.

The third configuration comprises two vertical columns usually round in shape, interconnected together at the lower part to form a U tube type connection.

Because of the U tube form, the force required to compact the resin during service cycle as well as during regeneration cycle is very small. Consequently low velocity is sufficient to compact the resin. The equation is based on the drag force applied by flow against particle.

Consider the U tube model of the present invention as shown in the following figure.



The fluid exerts a drag force F_d on the resin such that the resin column is displaced a distance h on each side of the tube.

Suppose the fluid flows from the right tube downward and upward to the other tube. The surface of the resin in the right tube is displaced down a distance h and in the left tube the surface of the resin is displaced up a distance h , so that the difference between the level of the resin on each tube is $2h$. The work required to move the resin column is $F_d h$. If the friction force is f_r , then the energy balance gives:

$(F_d h - f_r h) = \frac{1}{2} \rho_p A g h^2$ where the right hand side is the raise of the potential energy of the resin. Therefore

$$(F_d - f_r) = \frac{1}{2} \rho_p A g h \quad (1)$$

But the drag force is equal to

$$F_d = C_a \rho u^2 / 2g_c \quad (2)$$

where C is a constant, and ρ is the density of the fluid.

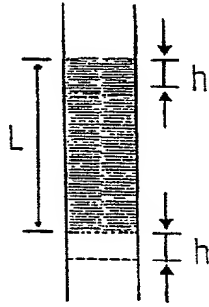
Combining Eqs.(1) and (2) yields:

$$u = \sqrt{(\rho_p g g_c h / C \rho + f_r \cdot 2 g_c / C A \rho)} \quad (3)$$

Now consider a conventional system with a resin column in a straight tube as shown below. The resin column is displaced a distance h to make it compact. Suppose the length of the resin column is L and its mass is m .

Energy balance equation yields:

$$(F_d - f_r - mgL)h = \frac{1}{2} \rho_p A g h^2 \quad (4)$$



Substituting $F_d = C A \rho u^2 / 2 g_c$ gives :

$$u = \sqrt{(\rho_p g g_c h / C \rho + f_r \cdot 2 g_c / C A \rho + 2 m g g_c L / C A \rho)} \quad (5)$$

Comparison of Eqs.(3) and (5) shows that the required velocity to make the resin compact in the conventional system is higher than that of the present invention.

It has been discovered that the resin bed remains compacted even when the cycle of the system is interrupted. This is because the resistance of the resin bed is higher than the gravitational force that would move the resin from its initial position due to the different surface level of the resin in both columns.

Compacted bed increases the performance of regeneration and service cycle. During regeneration a compacted bed will keep the resin bed firm so that the exchange of ions will be most effective with the highly regenerated zone at the regenerant inlet which is also the service outlet of the service cycle.

The system is self cleaning. During regeneration the fine broken particles which may migrate to the top of the resin bed will be carried away together with the regenerant waste. The fine particles in the other

compartment will be carried away during the rinsing process. Due to the self cleaning mechanism, in most cases no backwashing is required.

Nevertheless this system is equipped with internal backwashing system. After several hundreds of operation cycles broken resin particles of sizes larger than nozzle opening may accumulate which will increase the pressure drop above the acceptable limit. Backwashing can be conducted by adjusting the backwash flow rate to fluidize the resin bed and carry the fine particles away with the backwash waste.

Another advantage of this system compared to other cocurrent systems is that this system may use larger tank with larger volume of resin so that longer backwash interval can be obtained because this system is not affected by low service and regeneration flow rates.

Brief Description of Figures

The present invention will now be further described in the accompanying drawings, in which:

Fig.1 is a schematic representation of a currently available cocurrent system (the prior art)

Fig.2A is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the normal down flow service mode.

Fig.2B is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the regeneration mode.

Fig.3A is a schematic representation of a currently available counter current system with upflow service and down flow regeneration (the prior art) operating in the normal upflow service mode.

Fig.3B is a schematic representation of a currently available counter current system with upflow service and down flow regeneration (the prior art) operating in the regeneration mode.

Fig.4A is a schematic representation of separation system according to the present invention operating in the normal service mode.

Fig.4B is a schematic representation of separation system according to the present invention operating in the regeneration mode.

Fig.4C is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the left column.

Fig.4D is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the right column.

Fig.5 is a schematic representation of softening process according to the present invention operating in the service.

Fig.6 is a schematic representation of water demineralizing process according to the present invention using strong acid cationic resin for the cation column and strong base anionic resin for the anion column, operating in the service mode.

Fig.7 is a schematic representation of demineralizing and decolourization of sugar solution according to the present invention using strong acid cationic resin for the cation column and strong base macrorecteular anionic resin for the anion column, operating in the service mode.

Fig.8 is a schematic representation of water demineralizing process according to the present invention using combination of weak acid cation resin and strong acid cation resin for the cation column, degasifier, combination of weak base anion resin and strong base anion resin for the anion column with strong acid cation polisher and strong base anion polisher.

Fig.9A is a schematic side sectional view of one of the apparatus according to the present invention.

Fig.9B is a schematic top sectional view of one of the apparatus according to the present invention.

Fig.10A is a schematic side sectional view of one of the apparatus according to the present invention with an alternative design.

Fig.10B is a schematic top sectional view of one of the apparatus according to the present invention with an alternative design.

Fig.11A is a schematic side sectional view of one of the apparatus according to the present invention with another alternative design.

Fig.11B is a schematic top sectional view of one of the apparatus according to the present invention with another alternative design.

Complete description of invention

Figure 1 is a simplified schematic representation of a currently cocurrent system (the prior art). Column 1 with nozzle equipped underdrain bed 4 is filled with ion exchange resin about 50 % to 60 % of the total volume. Above the resin bed is freeboard 7. During service the liquid flows from port 2, which comes into contact with the compacted resin bed 5 and flows out to the outlet through port 3. During regeneration the direction of flow of the regenerant is identical to the service operation. Before the regeneration the resin is to be backwashed with the direction of flow from port 3 as the inlet to the outlet through port 2. This upflow direction will fluidize the resin bed 5, and resin fines and contaminated particles will be removed during this process. The resin level and backwash effectiveness can be monitored through sight glass 6.

Figure 2a and 2b are schematic representations of a currently available counter current system with down flow service and upflow regeneration (the prior art). Column 11 with nozzle equipped underdrain bed 14 and upper bed 15 is filled with ion exchange resin. During service (Figure 2a) the fluid is directed through port 12 passing the compacted resin bed 16a with outlet at port 13. During regeneration (Figure 2b) the regenerant passes through port 13 and the resin bed 16b with outflow through port 12. The resin will not be compacted and this will affect the regeneration efficiency. The level of the resin can be monitored from sight glass 18. Free board 17a and 17b are about 20 cm to 30 cm height just to allow the resin to expand during service operation. This system does not have an internal backwashing system. Therefore when the resin has to be backwashed it should be removed from the column to an external resin cleaning system.

Figures 3a and 3b are schematic representations of a currently available counter current system with upflow service and down flow regeneration (the prior art). Column 21 with nozzle equipped underdrain bed 24 and upper bed 25 is filled with ion exchange resin. During service (Figure 3a) the fluid flows through port 23, passes the compacted resin bed 26a and flows out through port 22. The service flow should be controlled at such a speed to maintain the resin bed in a compacted state so that the counter current regeneration profile of the resin will always be obtained. Lower speed or interrupting the flow will cause the resin not in a compacted

state and destroy the regeneration profile of the resin which will affect the quality of the treated liquid.

During regeneration (Figure 3b) the regenerant passes through port 22 and the resin bed 26b with outflow through port 23. In this cycle the resin bed is always compacted and the regeneration efficiency is high. The level of the resin can be monitored through sight glass 28. Free board 27a and 27b are of about 20 to 30 cm height to allow the resin to expand during the service operation. This system does not have an internal backwashing system. Therefore when the resin has to be backwashed, it should be removed to an external resin cleaning system.

Figures 4a, 4b, 4c and 4d are schematic representation systems according to the present invention. Column 101 is divided into two compartments by partition 110 where the lower part is interconnected such as to form a U tube type connection. Nozzle equipped upper bed 104 and 105 are located at the upper part of this column.

During service (Figure 4a) the fluid flows through port 102, passes the compacted resin 106b downflow and upflows through resin 106a with outlet at port 103. A low flow speed is sufficient to compact the resin bed and once the resin is compacted it will remain in a compacted state even if the flow speed is reduced or interrupted. Since the resin is always compacted the counter current regeneration profile will always be obtained which yields high quality of product liquid.

During regeneration (Figure 4b) the regenerant flows through port 103, passes the compacted resin 106d downflow and upflows through resin 106c with outlet at port 102. A low speed of regenerant flow is sufficient to compact the resin so that the regeneration efficiency is very high. The level of the resin can be monitored through sight glass 109. Free board 108a and 108b are of about 20 to 30 cm height just to allow the resin to expand during the service operation.

Since the system is self cleaning, backwashing to remove resin fines or contaminated particles is normally not required. If after many operation cycles the pressure drop increases such that its value becomes double, internal backwashing may be conducted. When backwashing the left column (Figure 4c), the flow is conveyed through port 103, passes the resin 106e downflow and upflows through resin 106f, then flows to the outlet through

port 111. The flow is controlled by means of flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow. When backwashing the right column, the flow goes through port 102, passes the resin 106g downflow and upflows through resin 106h, and flows out through port 112. The flow is controlled through flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow.

Alternatively, additional backwash port may be located at the bottom of the tube for the inlet of the backwash fluid. In this mode the backwashing process can be conducted for either one of the two columns or for both columns simultaneously. Backwashing the left column from the bottom inlet is conducted by closing port 112 so that the backwash fluid flows from the bottom inlet passing through the resin bed 106f and flows to the outlet through port 111. Backwashing the right column from the bottom inlet can be conducted by closing port 111 so that the backwash fluid flows from the bottom inlet passing through the resin bed 106h and flows out through port 112. Backwashing both columns simultaneously is conducted by opening ports 111 and 112 and flowing the backwash fluid through the bottom inlet port passing through the resin bed 106f (or 106g) and 106e (or 106h) and outflowing through ports 111 and 112.

The backwashing process is also applicable for modified U-tube of 180° angle (vertical straight tube) equipped with upper and lower bed with internal backwashing utilizing controllable backwash port located above the lower nozzle bed and outlet backwash port located below the upper nozzle bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings. Backwashing the resin is conducted by passing the backwash fluid with controlled flowrate through the inlet lower port located above the lower nozzle bed and outflowing through the backwash outlet port located below the upper nozzle bed.

For effective backwash cleaning port 111 and port 112 are to be equipped with resin trap or screen of 0.3 mm to 0.4 mm opening so that the fine particles may be driven out through this opening.

Figure 5 is a softening process of water using the system of the present invention. Tank 201 is filled with strong cation resin in sodium form 206a and 206b. Regenerant is sodium chlorine solution.

Figure 6 is a demineralizing process of water using the system of the present invention. Tank 301a is filled with strong acid cation resin 306ab and 306aa, while tank 301b is filled with strong base anion resin 306ba and 306bb. Regenerants for the strong acid cation resin and the strong base anion resin are hydrochloric acid solution and sodium hydroxide solution respectively.

The service flow is conveyed from port 302a, passes the strong acid cation resin 306ab downflow and upflows through cation resin 306aa, and flows out through port 303a. From this cation exchanger the decationized water flows through port 302b, passes the strong anion resin 306bb downflow, upflows through the resin 306ba and flows out through port 303b. The product is a demineralized water of high purity.

Figure 7 is a demineralizing and decolourization process of sugar solution using the system of the present invention. Tank 401a is filled with strong acid cation resin 406ab and 406aa while tank 401b is filled with strong base macrorecteclar anion resin which has the capability to adsorb colour of the sugar solution.

Regenerant for the strong acid cation resin is hydrochloric acid solution while for the macrorecteclar strong base anion resin the regenerant is sodium hydroxide solution.

The service flow is conveyed from port 402a, passes the strong cation resin 406ab downflow and upflows through resin 406aa, then outflows through port 403a. From this cation exchanger the decationized sugar solution flows through port 402b, passes the strong macrorecteclar anion resin 406bb downflow and upflows through resin 406ba, then outflows through port 403b. The product is a demineralized and decolourized sugar solution of high purity.

Figure 8 is a demineralizing process of water containing bicarbonate salt using the system of the present invention with degasifier and polisher. Cation exchanger tank 501a is filled with weak acid cation resin 506ab on the left compartment and strong acid cation resin 506aa on the right compartment. The use of these resin combination increases the regeneration efficiency. During regeneration with hydrochloric acid solution the regenerant flow is from port 503a downflow passing the strong cation resin 506aa. After compacting the resin the regenerant waste which still contains

hydrochloric acid at lower concentration passes through and regenerates weak acid cation resin 506ab upflow and outlets through port 502a.

Degasifier 513 contains deaerator fills 515 and equipped with air blower 514 which blows the air from the lower part of the fills upward.

5 Decationized water from the cation exchanger containing bicarbonate acid passes through the upper part of the fills and the carbon dioxide gas will be removed along with the blown air.

10 Anion exchanger tank 501b is filled with weak base anion resin 506bb in the left compartment and strong base anion resin 506ba in the right compartment. The use of this resin combination increases the regeneration efficiency.

15 During regeneration with sodium hydroxide solution the regenerant flow is from 503b downflow passing the strong anion resin 506ba. After compacting this resin the regenerant waste which still contain sodium hydroxide at lower concentration passes and regenerates the weak base anion resin 506bb upflow and outlets through port 502b.

20 Cation exchanger tank 501c is filled with strong cation exchanger 506cb and 506ca while anion exchanger tank 501d is filled with strong anion exchanger 506db and 506da. These two ion exchangers function as polisher and has the same design as the system in Figure 6. The service flow of the feed water which contains bicarbonate is conducted from port 502a, passes the weak acid cation resin 506ab downflow and upflows through the strong acid cation resin 506aa with outlet port at 503a. The decationized water which contains bicarbonate acid passes degasifier 513 and gets in contact with degasifier fills 515. The CO₂ gas from the bicarbonate acid is carried away by the blown air from blower 514. The degasified decationized water is pumped by pump 516 to the anion exchanger through port 502b , passes the weak base resin 506bb downflow and upflows through the strong base anion resin 506ba, and flows out through port 503b. The demineralized water is then polished in the cation polisher, passes port 502c and flows through strong cation resin 506cb downflow and upflows through resin 506ca with outlet at port 503c. From this cation polisher the water is then conveyed to the anion polisher through port 502d, passes the strong base anion resin 506db downflow and upflows through resin 506da, and goes outlet through port 503d. The

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treated water passing port 503b will have a high quality deionized water with conductivity up to less than 1 $\mu\text{S}/\text{cm}$ and residual silica up to less than 20 PPB and after passing through the cation and anion polisher with outlet at port 503d the conductivity can be less than 0.1 $\mu\text{S}/\text{cm}$ and residual silica less than 5 PPB.

Figures 9a to 11b illustrate the schematic sectional view of the apparatus using the present invention in three configurations. Figures 9a and 9b illustrate a configuration where the vertical column 101 is divided into two compartments by partition 110 with free space at the bottom part so that it forms a U tube type connection. At the upper part of the column is an upper bed with nozzles 104 and 105. Between the upper bed and the surface of the resin bed 106b is a free board 108a to accommodate the expansion of the resin bed after the resin has been exhausted, and also to accommodate resin expansion during backwash. The level of the resin bed can be observed through sight glass 109. Port 102 is an inlet of the service fluid and outlet for the regenerant fluid while port 103 is an outlet of the service fluid and inlet of the regenerant fluid. The operation can be reversed. Ports 111 and 112 are the outlets for the backwash fluid.

Figures 10a and 10b illustrate another configuration consisting of two vertical columns forming an U tube type connection. At the upper part of the columns 601a and 601b are upper beds equipped with nozzles. Between the upper bed and the surface of the resin bed 606b is a free board 608b to accommodate the expansion of the resin bed after the resin becomes exhausted as well as to accommodate the resin expansion during backwashing. The level of the resin bed can be observed through sight glass 609. Port 602 is an inlet of the service fluid and outlet of the regenerant fluid while port 603 is an outlet of the service fluid and inlet of regenerant fluid. The operation can be reversed. Ports 611 and 612 are the outlets for backwash fluid.

Figures 11a and 11b illustrate a configuration with a vertical column 701 and a smaller vertical column 701b at the inside with a connecting space at the bottom part so that the fluid as well as the resin can flow freely from one compartment to the other compartment. At the upper part of the columns is an upper bed with nozzles 704a and 704b. Between the upper bed and the surface of the resin bed 707a is a free board 708a to

accommodate resin expansion when the resin has been exhausted or during backwash. The level of the resin bed can be observed from the sight glass 709. Port 702 is an inlet of the service fluid and outlet of the regenerant fluid while port 713 is an outlet of the service fluid and inlet of the regenerant fluid. Port 703 is an alternative of port 702 which has the same function. Port 711 is the outlet for the backwash fluid. Port 712 is an alternative of port 711 which has the same function.

Example (Comparative data)

The chemical requirements, conductivity and silica leakage of a system according to the present invention are compared with those of a cocurrent system which are presently widely used. Each system is calculated with the same resin type, flow rate, water analysis and approximately the same resin quantity. Both systems are operating without degasifier.

Flow rate : 16.5 m³/h, net run 400 m³
 Water analysis (in m.eq) : HCO₃⁻ 0.334, CO₂ 0.334, SO₄⁻ 0.850, Cl⁻ 0.500, NO₃ 0.039, SiO₂ 0.194, Mg⁺⁺ 0.168, Ca⁺⁺ 0.997, Na 0.552.

	System of the present invention	Co-current system
Resin type	6000 l Amberjet 1200 4200 l Amberlite 96 RF 4500 l Amberjet 4200	6000 l Amberjet 1200 4500 l Amberlite 96 RF 4500 Amberjet 4200
Regenerant consumption	33 kg HCl (As 100%) 36 kg NaOH (100%)	53 kg HCl (As 100%) 36 kg NaOH (100%)
Tank diameter	1000 mm	1000 mm
Effective tank height	1700 mm	3100 mm
Conductivity	1.0/3.0 µS/cm	3.0/6.0 µS/cm
Average/end point		
Silica leakage	0.03/0.10 mg/l	0.12/0.20
Average/end point		

From the data mentioned above it can be observed that the system

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Claims

1. An ion exchange process using U-tube principle consisting of two connected compartments with internal backwashing utilizing controllable backwashing ports located below the nozzle bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings.
2. An ion exchange process according to claim 1 in which the backwash process is conducted only if the pressure drop of the service flow increases significantly, backwashing the resin at the second compartment is conducted by passing the backwash fluid with controlled flowrate through the inlet port at the first compartment and outflowing through the backwash outlet port located below the nozzle bed, backwashing the resin at the first compartment is conducted by passing the backwash fluid with controlled flowrate through the outlet port at the second compartment and outflowing through the backwash outlet port located below the nozzle bed.
3. An ion exchange process according to claim 1 in which the backwash process is considered only if the pressure drop of the service flow increases significantly; backwashing process is conducted by passing the backwash fluid through the inlet port located at the bottom of the U-type tube consisting of two connected compartments and outflowing through the outlet ports located below the upper nozzle beds in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings;
backwashing the resin in the first compartment is conducted by closing the backwash port below the upper nozzle bed of the second compartment and flowing the backwash fluid through the bottom inlet passing the resin bed in the first compartment and outflowing through the backwash port located below the upper nozzle bed of the first compartment; backwashing the resin in the second compartment is conducted by closing the backwash port below the upper nozzle bed of the first compartment and flowing the

backwash fluid through the bottom inlet passing the resin bed in the second compartment and outflowing through the backwash port located below the upper nozzle bed of the second compartment; simultaneous backwashing of both compartments is conducted by opening both backwash ports of the first and second compartment and flowing the backwash fluid through the bottom inlet passing the resin beds in the first and second compartments and outflowing through backwash ports below the nozzle beds of the first and second compartments.

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4. An ion exchange process using modified U-tube of 180° angle (vertical straight tube) equipped with upper and lower bed with internal backwashing utilizing a controllable backwash port located above the lower nozzle bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings.

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5. An ion exchange process according to claim 4 in which the backwash process is conducted only if the pressure drop of the service flow increases significantly; backwashing the resin is conducted by passing the backwash fluid with controlled flowrate through the lower port located above the lower nozzle bed and outflowing through the backwash outlet port located below the upper nozzle bed.

20

Abstract

AN ION EXCHANGE SYSTEM WITH COMPACTED MEDIA
DURING OPERATION AND REGENERATION
PROCESSES USING U-TUBE PRINCIPLE

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10 This invention is a liquid separation system using ion exchange resin, adsorbent resin and catalytic resin based on U-tube principle. It is shown that relatively low fluid velocity is sufficient to maintain the resin bed in compacted state during the operation as well as during the regeneration processes so that high quality product, low regenerant consumption, and convenient operation are obtained.

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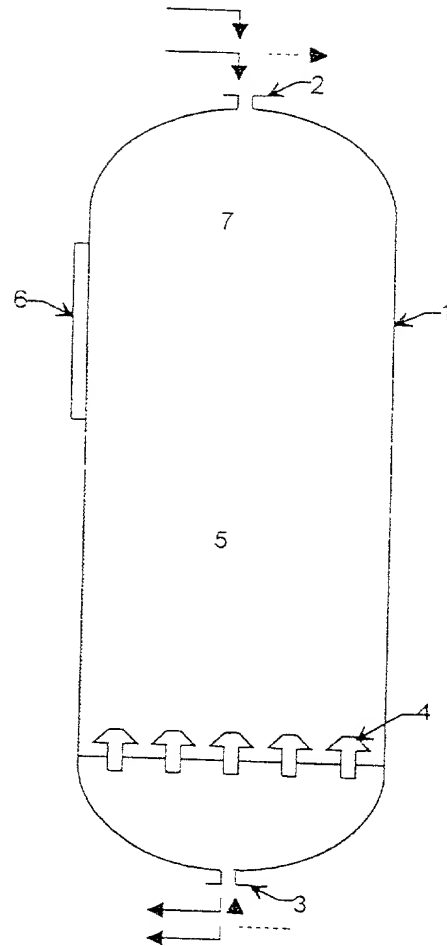


Fig.2a

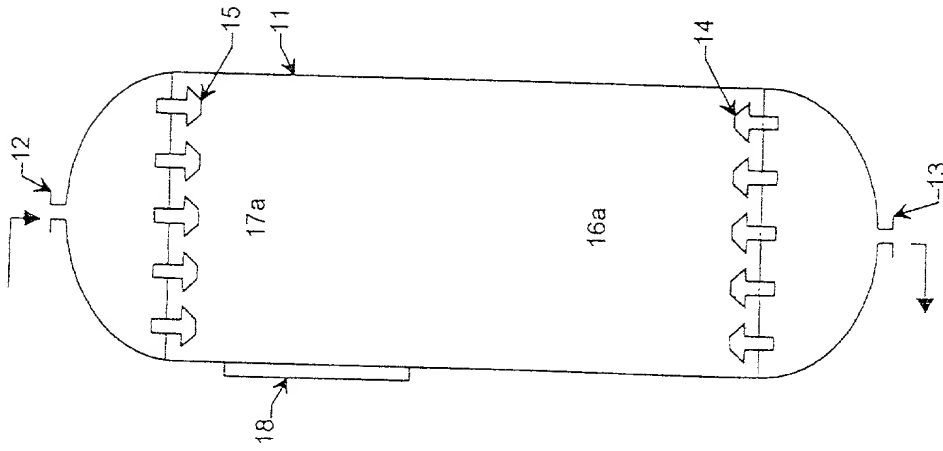


Fig.2b

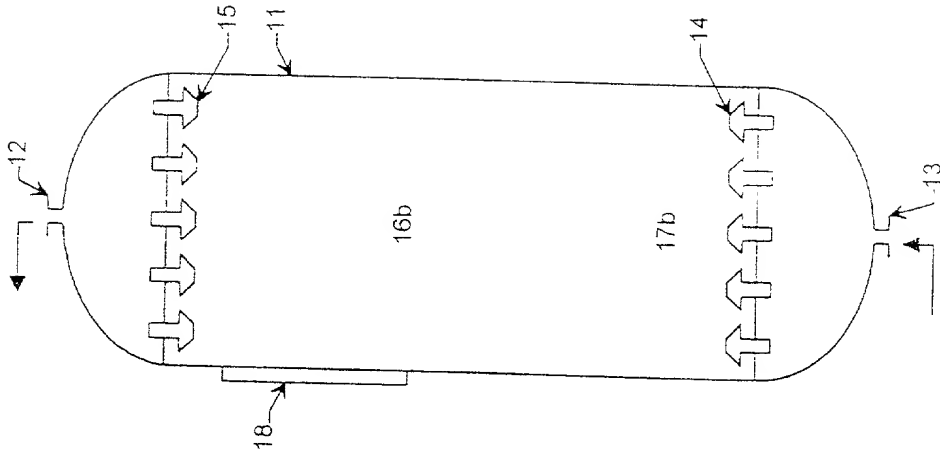


Fig. 3a

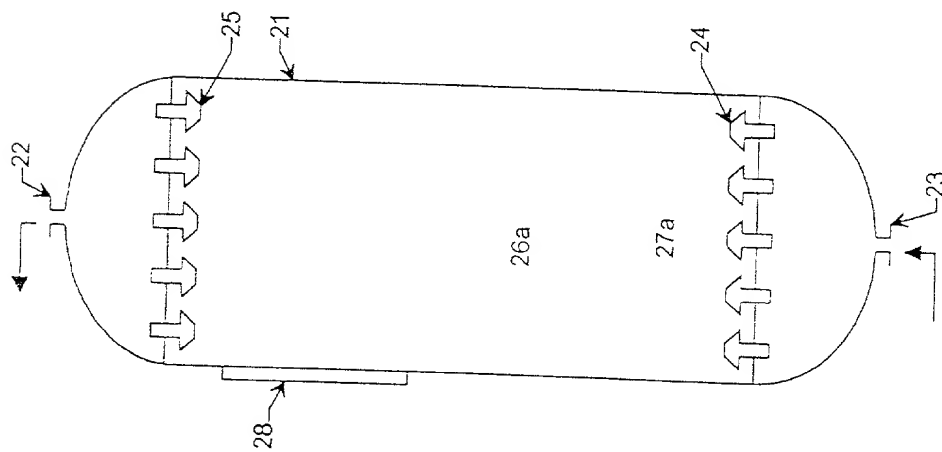
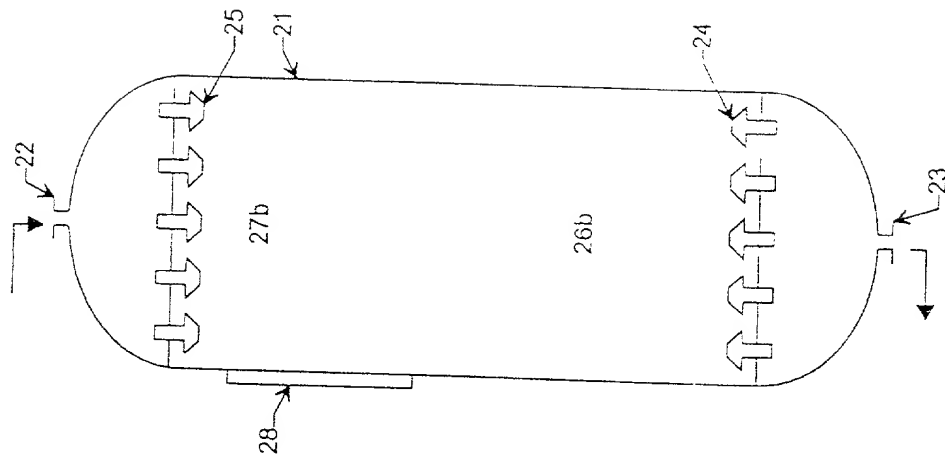
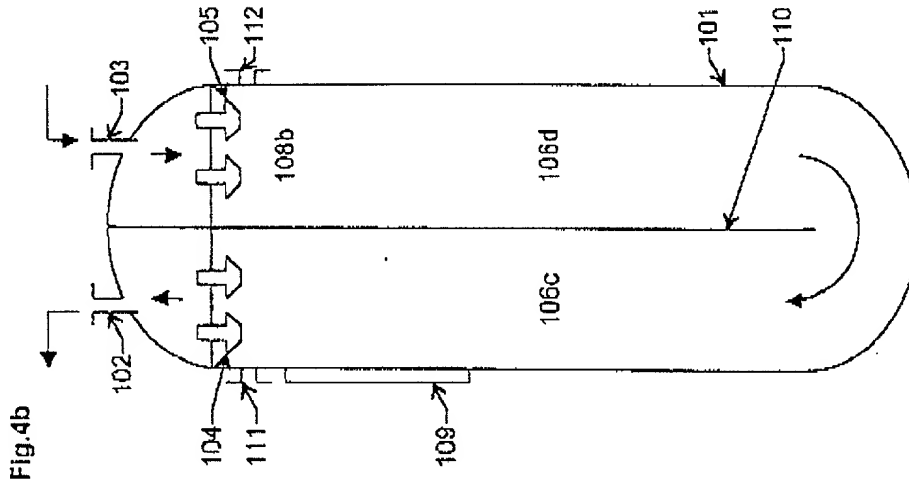
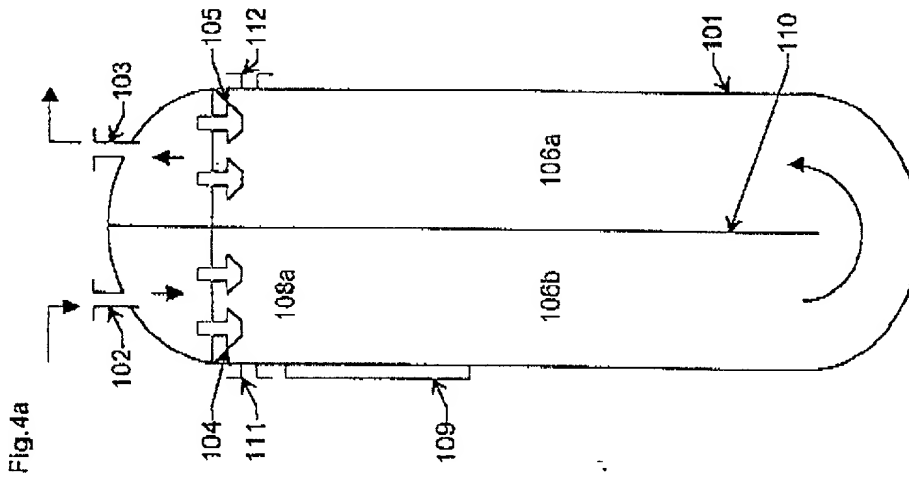


Fig. 3b





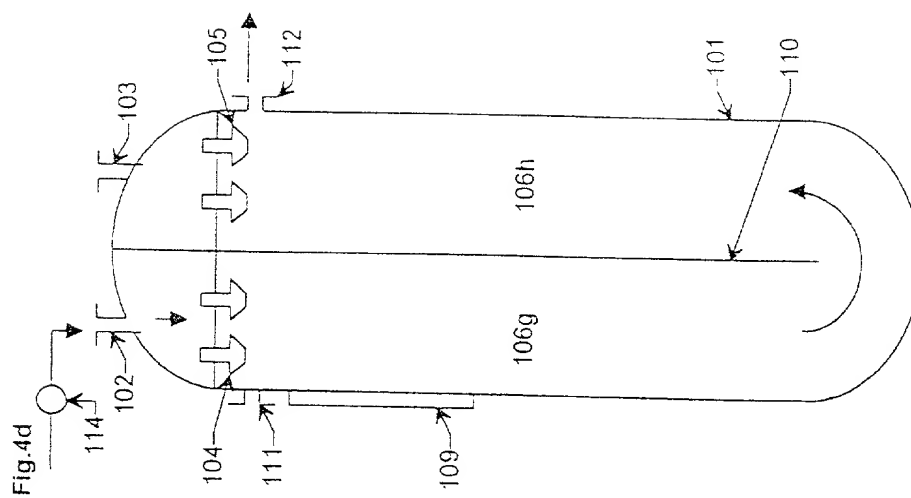
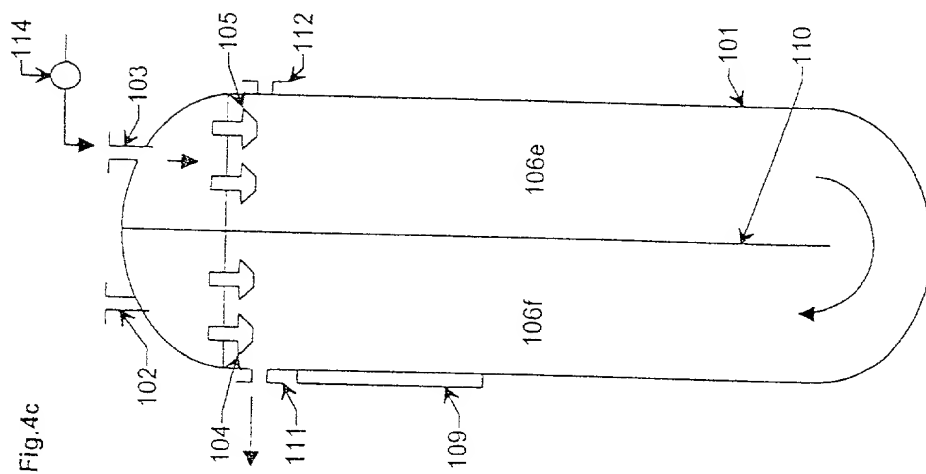


Fig.5

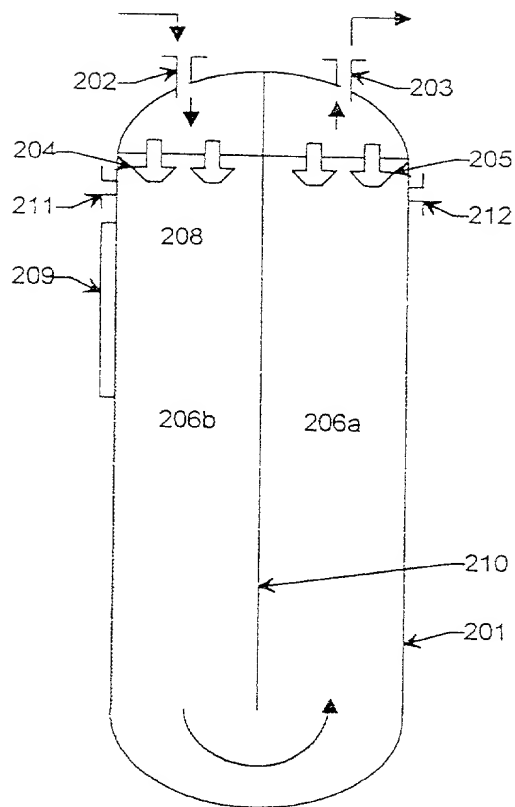
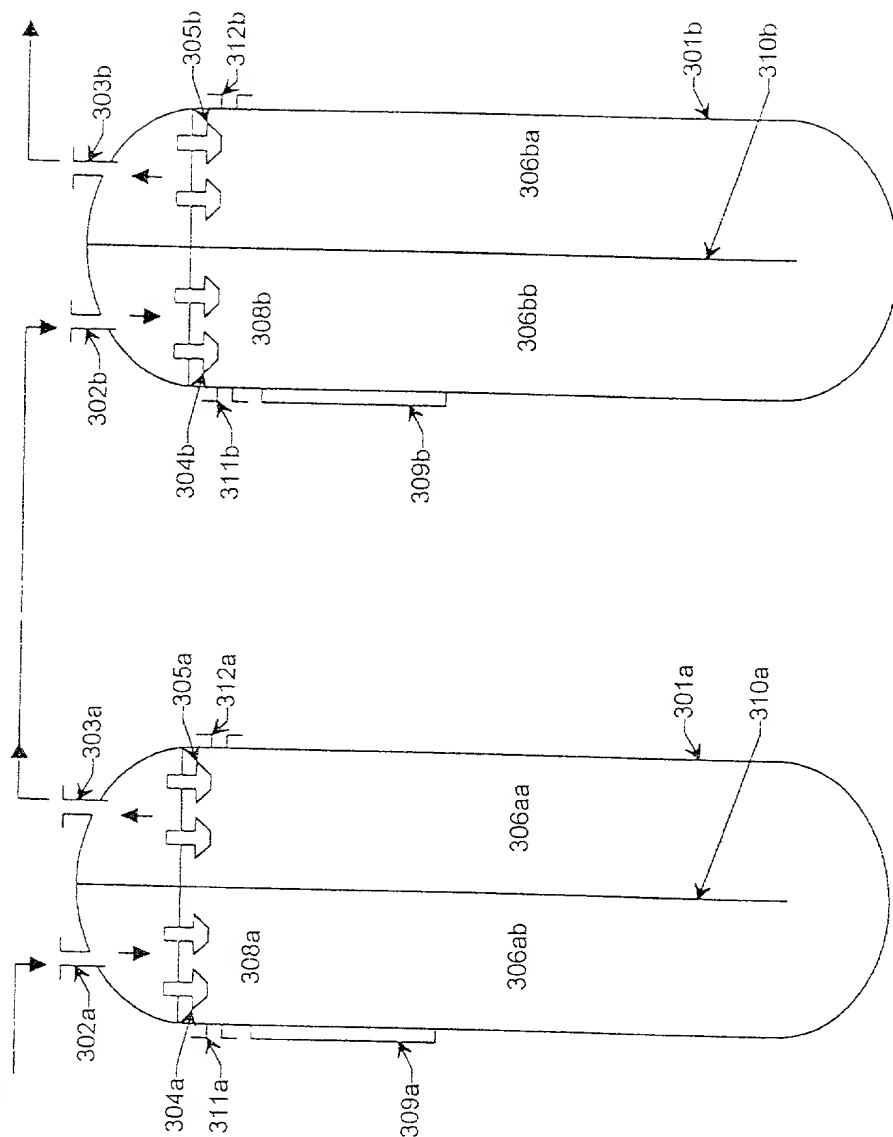


Fig.6



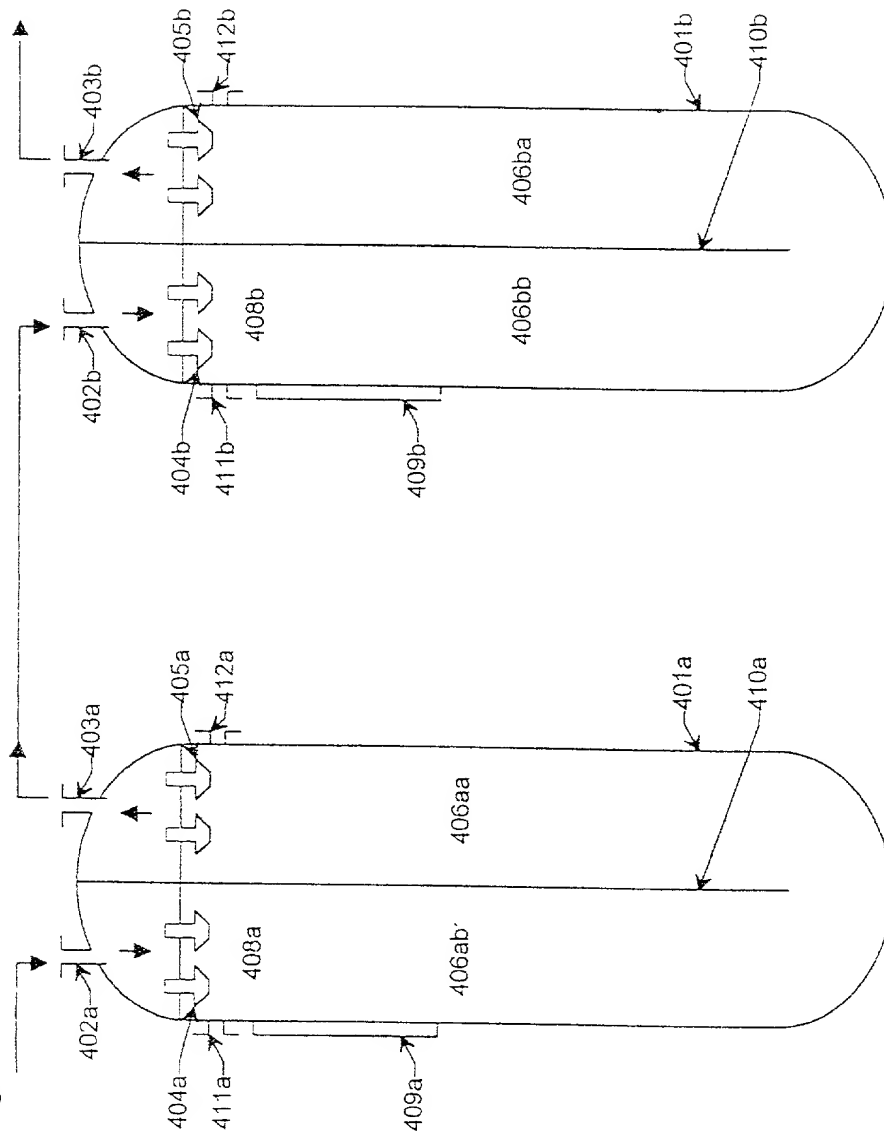


Fig.8

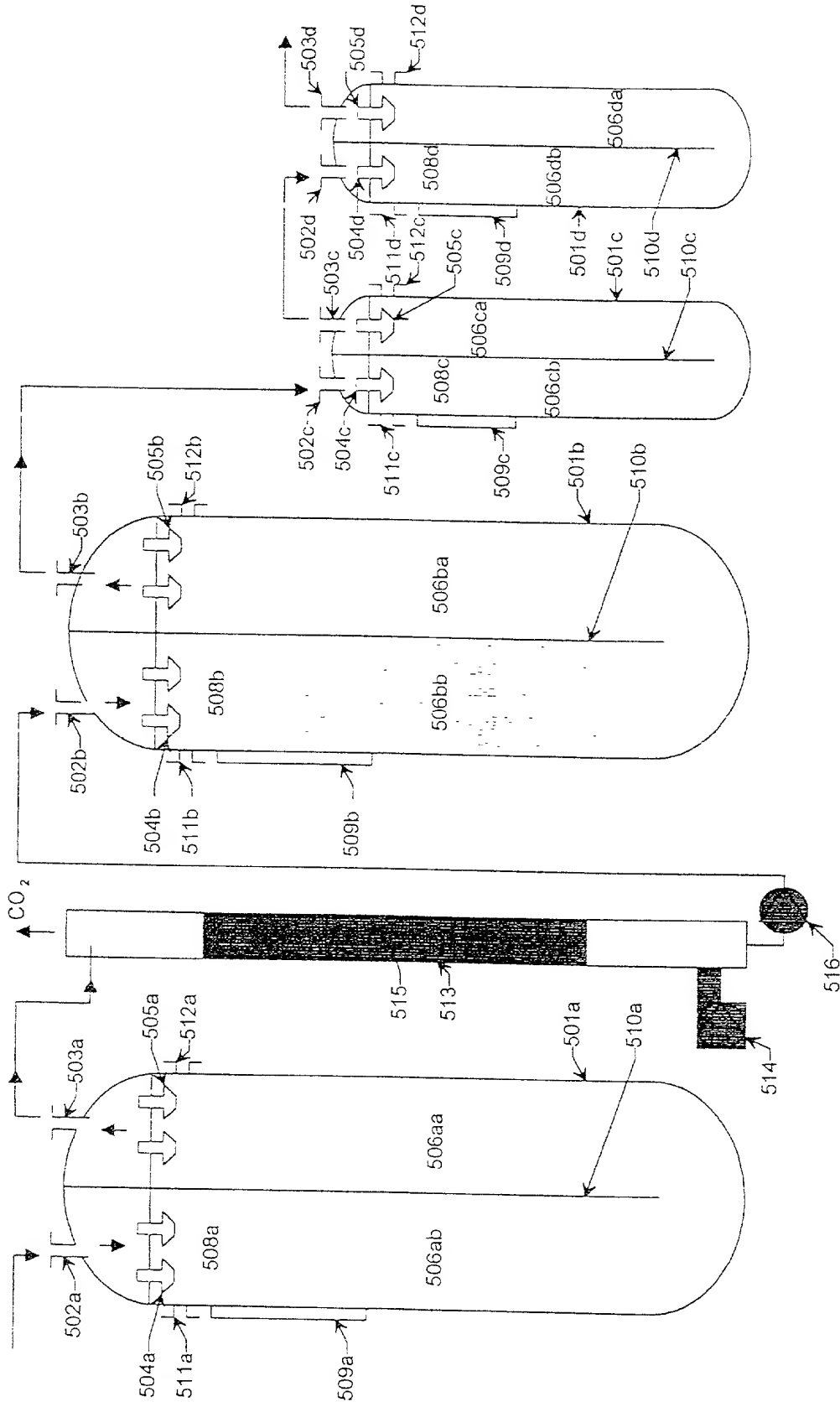


Fig.9a

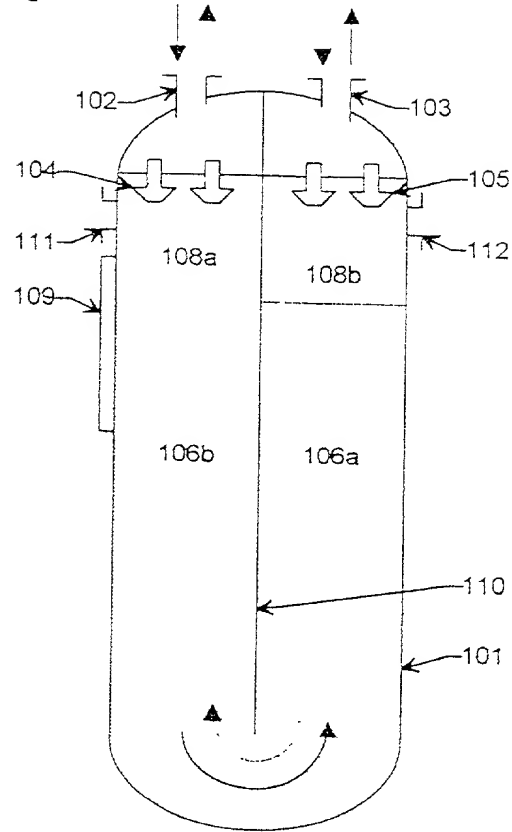


Fig.9b

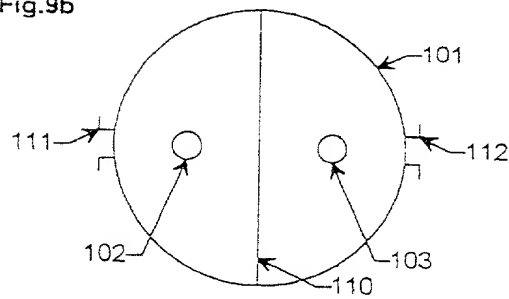


Fig.10a

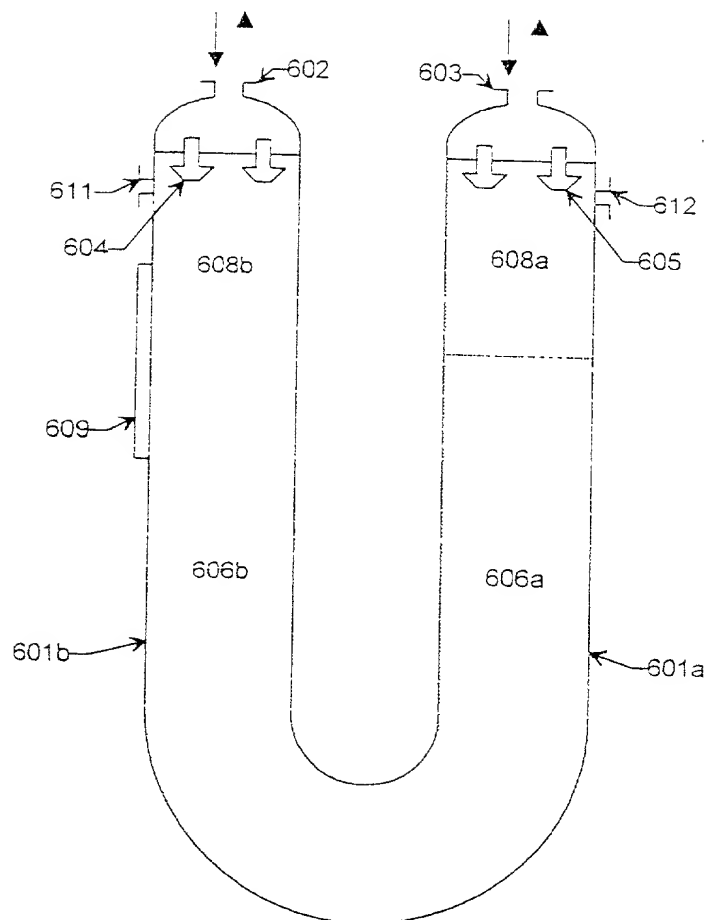


Fig.10b

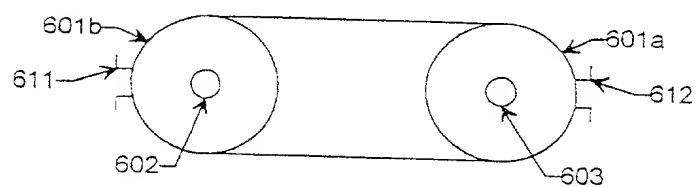


Fig.11a

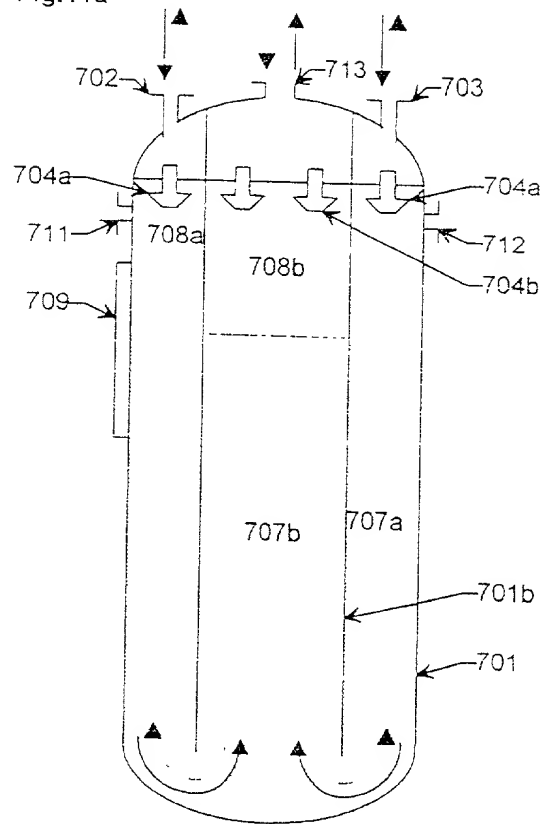
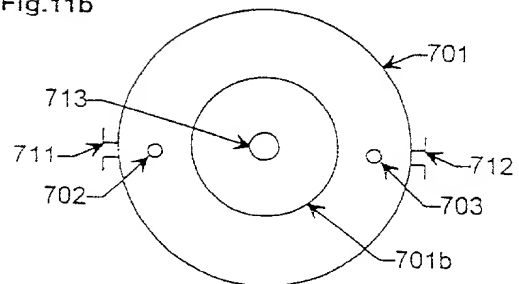


Fig.11b



Rev 12/99

DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATIONS

PATENT

Docket No. 39629/DBP/R130

As an inventor, I declare

My residence address and citizenship are as stated next to my name. I believe I am the original, first and sole inventor (if only one name is listed below), or a joint inventor (if plural names are listed below) of the invention claimed in the patent application entitled

(1) ION EXCHANGE SYSTEMS USING U-TUBE PRINCIPLE

the specification of which

(2) X was filed on 7 July 1998 as United States Application Number of PCT International Application
(3) Number PCT/IB98/01039 and was amended on _____ (if applicable)

I have reviewed and understand the contents of the specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with 37 CFR § 1.56.

(4) X I hereby claim the benefit under Title 35, U.S.C. § 119(e) of any United States provisional application(s) listed belowP-97395323 December 1997

Application Serial Number

Filing Date

Application Serial Number

Filing Date

(5) X I claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) of the foreign application(s) for patent or inventor's certificate listed below, and I have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Claimed

P-973593Indonesia23 December 1997Yes

Number

Country

Day/Month/Year

Yes

No

Number

Country

Day/Month/Year

Yes

No

(6) I claim the benefit under 35 U.S.C. § 120 of any United States application(s) listed below, and insofar as this application discloses and claims subject matter not disclosed in the prior United States application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose material information as defined in 37 CFR § 1.56 which occurred between the filing date of the prior application and the national or Patent Cooperation Treaty International filing date of this application

Application Serial Number

Filing Date

Patented/Pending/Abandoned

Application Serial Number

Filing Date

Patented/Pending/Abandoned

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon

(7) **POWER OF ATTORNEY:** I appoint the following attorneys and agents of the law firm CHRISTIE, PARKER & HALE, LLP to prosecute this application and any international application under the Patent Cooperation Treaty based on it and to transact all business in the U.S. Patent and Trademark Office connected with either of them in accordance with instructions from the assignee of the entire interest in this application; or from the first or sole inventor named below in the event the application is not assigned; or from BIRD OKTROI ROOSSENSO in the event the power granted herein is for an application filed on behalf of a foreign attorney or agent

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Rev 12/99


DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATIONS

1947-1948

Docket No. 39629/DBP/R130

Docket No.

(8)

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Residence and Postal Office Address	Examination	

Full Name of Third or Joint Inventor	Inventor's Signature	Date
Residence and Post Office Address		1917-1918

Full Name of Fourth or Joint Inventor	Inventor's Signature	Date
Residence and Post Office Address	Witnesses	

Full Name of Editor (not in code)	Inventor's Signature	Date
Residence and Post Office Address	City and State	

Full Name of Sixth or Joint Inventor	Inventor's Signature	Date
Residence and Post Office Address	City and State	

Full Name of Inventor or Joint Inventor	Inventor's Signature	Date
Residence and Post Office Address		Witnessing

Full Name of Eighth or Joint Inventor	Inventors Signature	Date
Residence and Post Office Address		Relationship

U.S. DEPARTMENT OF AGRICULTURE, WASHINGTON, D.C.

-2-

TOTAL P.03